UCRL- 90097
PREPRINT

A COMPARISON OF THE OUTGASSING CHARACTERISTICS OF SEVERAL SOLAR ABSORBING COATINGS

E.D. Erikson
D.D. Berger
B.A. Frazier

This paper was presented for submittal to the
Journal for
Vacuum Sciences & Technology

April 3, 1985

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ABSTRACT

An investigation has been conducted of the room temperature outgassing characteristics of several optically black solar absorbing coatings. The coatings were deposited either electrochemically, by spraying, or by brushing onto aluminum or stainless steel substrates. The vacuum outgassing rates and residual gas evolution have been studied for exposure times on the order of 100 hours. Representative outgassing values are given for the following coating/substrate combinations: anodized and black dyed aluminum, etched electroless nickel on aluminum, black chrome on aluminum and on stainless steel, and black paint on aluminum. The results confirm that the outgassing rates of these coatings can be expressed as the exponential decay function [1] of the form $q_t = At^{-m}$, where the outgassing rate at a specific time, q_t, is a product of the outgassing rate after one hour exposure to vacuum, A, and the time t, raised to the inverse power of m. In the case of the anodized aluminum, m was found to be approximately equal to 0.5. For the other materials m was found to be about equal to 1.0.

I. INTRODUCTION

Outgassing characterization studies of several solar absorbing coatings were conducted. The coatings have high absorptivity in and near the visible light region and low emissivity at the longer infrared wavelengths [2]. Possible applications of these coatings include solar energy conversion, plasma physics experiments, and optical transport systems.

The following coatings will be discussed: A) Aluminum, anodized and dyed black; B) Electroless nickel on aluminum, which has been acid etched to produce a black coating; C) Black chrome on aluminum; D) Black chrome on stainless steel; E) 3M ECP-2200 black paint on aluminum. In each case, the substrates consisted of 0.328 cm x 12.7 cm x 25.4 cm type 304 stainless steel or type 6061-T6 aluminum.

II. EXPERIMENTAL

A. OUTGASSING TEST SYSTEM

The system used for performing the outgassing characterization tests is shown in Fig. 1. The 304 stainless steel chamber has a volume of 11.2 l and an internal surface area of 3900 cm^2. The 0.457 mm diameter orifice (item 3) has a calculated conductance of 2×10^{-2} l/s for N_2 at room temperature. The procedures used during testing and the methods used to reduce the outgassing and residual gas data have been published[3,4].

The outgassing of the test chamber was found to be consistent from run to run. The outgassing rate as a function of time is in agreement with those data presented by Strausser [5] for 304 stainless steel. The gas contribution from the test chamber (outgassing rate and residual gas spectra) was usually negligible when compared to that from the material being tested.

B. SAMPLE PREPARATION

1. <u>Black Anodized Aluminum.</u>

Aluminum substrates were cleaned and sulfuric acid anodized according to a process described by Wodehouse[6]. They were then rinsed, immersed in an organic black dye solution[7] and sealed with a nickel acetate solution[8].

2. <u>Black Etched Electroless Nickel on Aluminum.</u>

The substrates were cleaned as described above. They were then double zincate treated[9], electroless nickel coated with a nickel-phosphorous alloy[10] and processed in a 50 volume percent nitric acid solution using a procedure described by Johnson[11]. Two features of this coating are considered unique[12]: electroless nickel can be plated onto virtually any substrate; and the blackening results from the etching of conical light traps into the nickel, avoiding the use of any dyes.

3. <u>Black Chrome on Aluminum.</u>

The substrates were pre-cleaned and zincate treated as described above. After treatment, they were rinsed in distilled water and electroplated in a Rochelle salt copper strike solution[13] to give an adhesive layer on which to bond the chrome. After the copper strike, the substrates were plated in a proprietary black chrome solution[14].

4. <u>Black Chrome on Stainless Steel.</u>

Type 304 stainless steel substrates were cleaned, acid pickled, given a Wood's nickel strike[15] and then plated with black chrome to a coating thickness of approximately 220 nm. More complete details of the preparation and plating processes are given in Beat, et al.[16].

5. <u>Black Paint, Brushed onto Aluminum.</u>

One of the many commercially available black paints was investigated as an alternative to electroplated coatings. The paint, 3M brand ECP-2200 Solar Absorber Coating, is said to have a solar absorption of 0.96. It is unaffected by temperatures up to 540°C (1000°F)[17], and has been the subject of study for possible aerospace applications[18]. The paint was applied by brushing, and was air dried for 72 h at ambient temperature before testing.

6. <u>Black Paint, Sprayed onto Aluminum.</u>

A second test of the 3M paint was made in order to determine the effect of a different method of application as well as to determine the effects of elevated temperature on the outgassing rate and gas spectra. The paint was sprayed onto aluminum substrates and air dried for 72 h prior to testing. The test included three temperature variations. First, room temperature outgassing for 24 h allowed comparison between application techniques. Next, the sample chamber which contained the painted substrates was heated to a temperature of 100°C and maintained at that temperature for 24 h. Finally, the test chamber and samples were allowed to cool to room temperature in order to determine the reduction in outgassing the rate and evolved gases which might result from bakeout.

III. RESULTS AND DISCUSSION

A. Black Anodized Aluminum.

The outgassing rate of the anodized and black dyed aluminum samples was found to be greater than the rate for anodized (but undyed) aluminum[19,20]. The outgassing rate of the former after one hour was $q = 4.2\times10^{-6}$ Torr l $s^{-1}cm^{-2}$ (5.6×10^{-3} Pa m s^{-1}), and the slope of the outgassing rate vs time curve was on the order of -0.7. The evolution of H_2 was found to increase from 20% to 30% of the total gas load over the 100 h test, while H_2O decreased from 70% to 60% of the total in the same period. Ammonia, which contributed approximately 10% to the total, and CO/N_2, which accounted for less than 1%, remained constant over the same 100 h period.

Although our outgassing tests only studied the material behavior during the first pumpdown of "as received" samples, other tests indicate that anodized aluminum will readily readsorb atmospheric contaminants. Heslin and Hunkeler[21], while characterizing various aluminum anodizes for spacecraft applications, found that the weight loss from any given anodized sample varied from 4 to 35 mg/cm^2 per mm of coating thickness when exposed to vacuum for 24 h. They determined that most of the weight loss is recovered once the sample is exposed again to atmosphere. Mass spectrometer analysis of scrapings from the coatings confirmed a predominance of water and absorbed nitrogen in anodized samples.

B. Black Etched Electroless Nickel on Aluminum.

After one hour under vacuum, the outgassing rate of the black electroless nickel was about 1.8×10^{-7} Torr l $s^{-1}cm^{-2}$ (2.4×10^{-4} Pa m s^{-1}). The slope of the outgassing curve was about -1.1. H_2 evolution was relatively constant at 35% to 38% of the total gas load during the initial 16 h of testing. Mass 28 (CO/N_2) increased from 2% to 26%, NH_3 decreased from 6% to a trace amount, H_2O decreased from 50% to 32%, and CO_2 remained constant at 4% to 5% over the same 16 h period. Due to an unusual amount of contamination from an ion gauge, residual gas analysis for all but the first 16 h of the run was unreliable.

C. Black Chrome on Aluminum.

After one hour of testing, the outgassing rate of aluminum coated with black chrome was about 4.6×10^{-6} Torr l $s^{-1}cm^{-2}$ (6.1×10^{-3} Pa m s^{-1}). The graph of the outgassing curve yielded a slope of approximately -1.6. The gas evolution of H_2 and NH_3 increased from 8% to 27% and from 8% to 10% respectively, over the initial 95 h. Over the same period, H_2O was found to decrease from 83% to 60% of the total gas load. Mass 28 (CO/N_2) evolution amounted to 1% or less throughout the entire test.

D. Black Chrome on Stainless Steel.

The outgassing rate of the black chrome coated stainless steel samples was about 9.0×10^{-7} Torr l $s^{-1}cm^{-2}$ (1.2×10^{-3} Pa m s^{-1}) after one hour, and the slope of the outgassing curve was about -1.2. Measurement of the residual gases during the 85 h test indicated that the evolution of H_2 increased slightly (from 26% to 32%), as did CO_2 (from a trace at the beginning of the run to 3% at the end). Mass 28 (CO/N_2) increased markedly from 1% to 18% while NH_3 and H_2O decreased from 7% to 3% and from 66% to 44%, respectively.

E. Black Paint, Brushed onto Aluminum.

The outgassing rate after one hour was determined to be 1.8×10^{-7} Torr l $s^{-1} cm^{-2}$ (2.5×10^{-4} Pa m s^{-1}) and the slope of the outgassing curve was about -1.0. Over the 100 h test, the outgassing rate of the black paint was nearly the same as that of the black electroless nickel samples. Evolved H_2 increased slightly from 26% of the total gas load to 31% of the total over the 100 h test. Methane increased from a trace to 1.2%, and CO/N_2 increased from a trace to about 24% during the test. Ammonia and H_2O decreased from 6% to a trace amount and from 42% to 11%, respectively.

F. Black Paint, Sprayed onto Aluminum.

The room temperature outgassing of the spray painted samples was found to be identical to that of the samples painted by brush. After 24 h, the test chamber containing the samples was heated to 100°C during a one hour period. The percentages of evolved gases and the outgassing rates measured for the spray painted black samples are given in Table I as a function of both temperature history and total time under vacuum.

IV. SUMMARY

The outgassing data for the samples of solar absorbing coatings which were tested are compared in Fig. 3. The coating which was least vacuum compatible, black anodized aluminum, has an outgassing rate approximately 10 times that of the most compatible, electroless nickel on aluminum. The solar absorbing coatings of black chrome and black paint were judged to have vacuum compatibilities which lie between these two extremes.

Surprisingly, the "as received" 3M ECP-2200 solar absorbing coating had a low outgassing rate - similar to that of the electroless nickel on aluminum. By using the a relatively low temperature bakeout described in the text, the outgassing rate of the paint can be reduced by an order of magnitude.

V. ACKNOWLEDGEMENTS

The authors would like to thank Mr. Wendell Kelley of the LLNL Coating Processes Group and Mr. Tom Beat of the LLNL Vacuum Processes Group for their help in this work. We would also like to thank Mr. Robert Bathgate and Mr. Gil Cruz, both of LLNL, and Mr. Brent Davis of EG&G, North Las Vegas, who expressed a need for this information and provided financial support for these tests.

AUSPICES

Work performed under the auspices of the United States Department of Energy and by the Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.

Table I. Evolved gases (expressed as a percentage of the total gas load) and outgassing rates from 3M ECP-2200 Solar Absorbing Coating as a function of the temperature and time under vacuum.

GAS SPECIES	Ambient Temperature (24 hrs.)	After Heating To 100°C (25 hrs.)	After 24 hrs At 100°C (49 hrs.)	After Cooling To 20°C (100 hrs.)
H_2	70.1	80.5	70.0	44.5
NH_3	0.7	0.7	1.0	0.9
H_2O	7.3	5.8	1.9	13.1
CO/N_2	15.6	9.1	23.3	28.6
CO_2	2.6	2.5	2.2	8.9
C_xH_y	3.6	1.5	1.8	4.1
OUTGASSING RATE. (Torr l $s^{-1}cm^2$)	8.2×10^{-9}	4.0×10^{-7}	1.3×10^{-10}	8.6×10^{-11}

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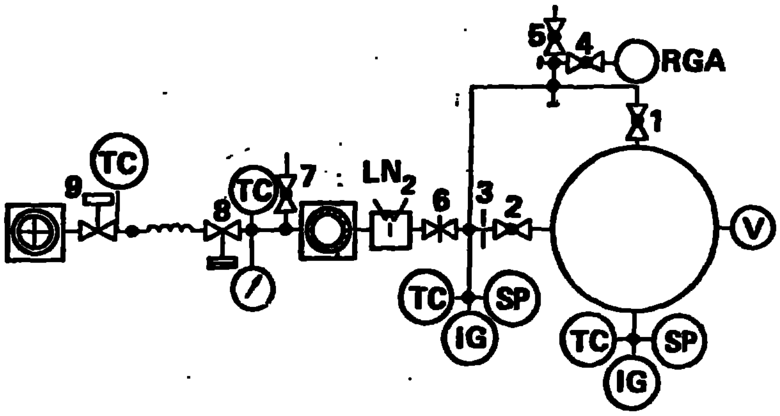

Figure 1. Schematic of the outgassing system used in determining the outgassing rate of materials[3]. Legend: (1), (2), (4-7) - manually-actuated valves; (3)-fixed orifice with conductance of $2x10^{-2}$ l/s for N_2 gas; (8) and (9) - pneumatically-actuated valves; (TC), (SP), (V) and (IG) - Convectron, Schultz-Phelps, Viscosity, and Bayard-Alpert vacuum gauges, respectively.

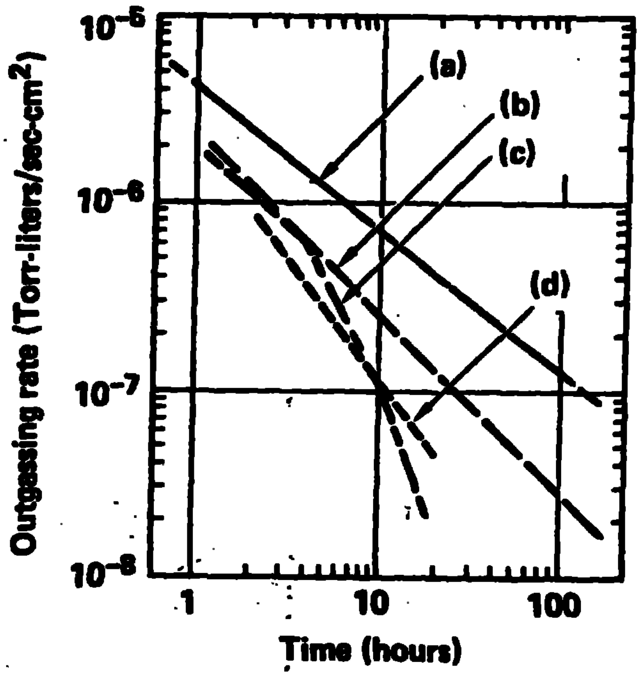

Figure 2. Comparison of various studies of the outgassing of anodized aluminum: a) Anodized and Black Dyed Aluminum, b) Clear Anodized Aluminum, LLNL[2], c) Clear Anodized Aluminum after Schram[10], d) Clear Anodized Aluminum after Blears, et al.[9].

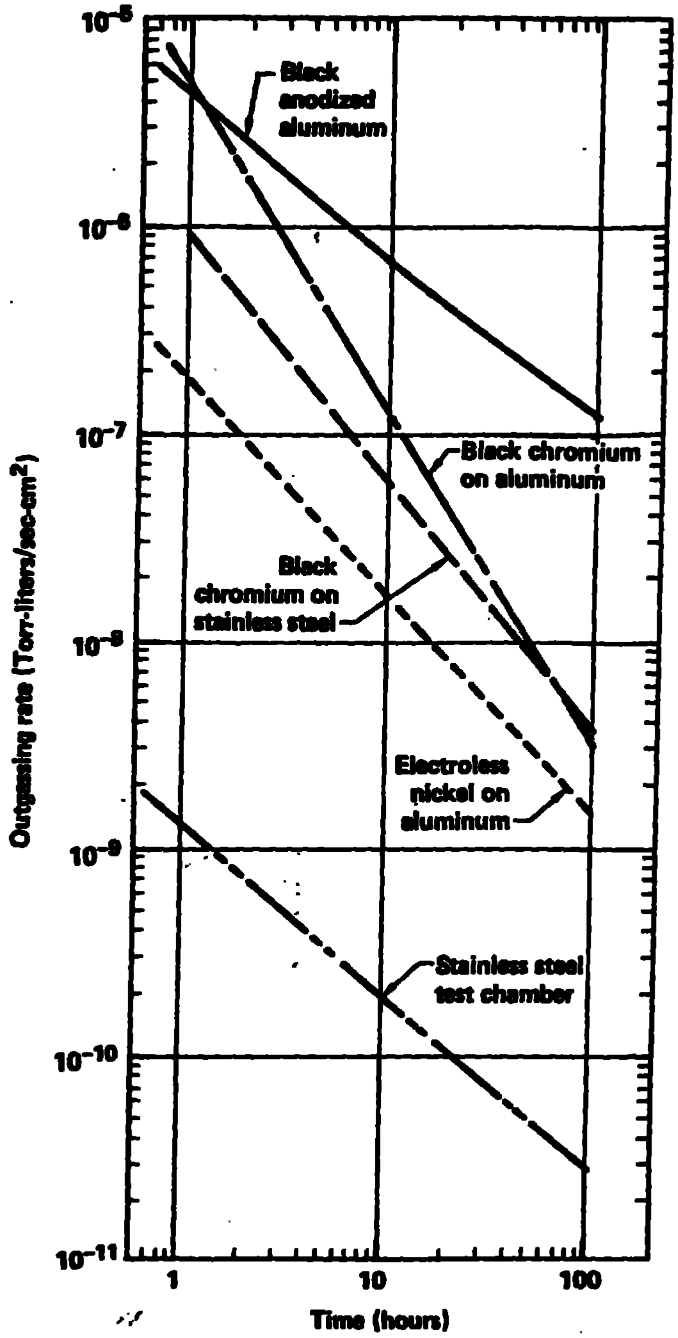

Figure 3. Comparison of the outgassing rates of the stainless steel test chamber and the black coatings tested. The room temperature outgassing of the black paint was found to be identical to that of black electroless nickel.